THE ANALYST.

DECEMBER, 1902.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

The monthly meeting of the Society was held on Wednesday evening, November 5, in the Chemical Society's Rooms, Burlington House. The President, Dr. J. Augustus Voelcker, M.A., B.Sc., occupied the chair.

The minutes of the previous meeting were read and confirmed.

A certificate of proposal for election to membership in favour of Mr. P. Macdonald was read for the second time; and certificates in favour of Messrs. Alexander Hutcheson Bennett, analytical chemist, 39, Lime Street, London, E.C.; James Charles McWalter, M.A. (R.U.I.), D.P.H. (Dublin), analytical chemist, 19, North Earl Street, Dublin; and William Partridge, A.I.C., 2, Ethelbert Road, Wimbledon, assistant to Mr. William Chattaway, F.I.C., were read for the first time.

The following papers were read: "The Reactions of the Alkaloids of Ipecacuanha," by Alfred H. Allen and G. E. Scott-Smith; "The Analysis of Preparations containing Opium," by Alfred H. Allen and G. E. Scott-Smith; "Note on the Estimation of Salicylic Acid," by Sidney Harvey; and a "Note on the Volatility of Aqueous Solutions of Acetic Acid," by William Chattaway.

CERTAIN REACTIONS OF THE ALKALOIDS OF IPECACUANHA.

By Alfred H. Allen and G. E. Scott-Smith.

(*Read at the Meeting, November* 5, 1902.)

Our attention was recently called to the fact that the alkaloids of ipecacuanha in some of their colour-reactions presented a considerable similarity to morphine, and we have made a number of experiments with the view of ascertaining to which of the ipecacuanha alkaloids these colour-reactions were attributable.

The best recent work on the alkaloids of ipecacuanha is that of Dr. B. H. Paul and A. J. Cownley. They have recognised, and isolated from ipecacuanha root, three alkaloids — namely, emetine ($C_{30}H_{44}N_2O_4$), cephaëline ($C_{28}H_{40}N_2O_4$), and psychotrine—to the last of which no formula has hitherto been assigned. It does not, however, follow that these are the only alkaloids present in ipecacuanha root, and in the opinion of a high authority there are at least five such bodies present. However, our experiments have been practically limited to the three alkaloids recognised by Paul and Cownley.

Of these alkaloids of ipecacuanha emetine is present in the largest amount, but a considerable proportion of cephaëline is also present. Psychotrine occurs in much smaller quantity. All three alkaloids are soluble in amylic alcohol. Emetine, which appears to be an amorphous alkaloid, yields crystallizable salts. Cephaëline crystallizes well, and psychotrine also forms highly characteristic crystals when properly treated. Emetine and cephaëline, when freshly precipitated, differ from psychotrine in being readily dissolved by ether after adding excess of ammonia to their salts, whereas psychotrine is not so extracted, but may be dissolved by subsequently agitating the ammoniacal liquid with chloroform.* Dry ether has little solvent action on dry emetine or cephaëline. Emetine and cephaëline are separated by Paul and Cownley by adding excess of caustic soda to the solution of their salts and agitating with ether, which takes up the emetine only. In this behaviour cephaëline closely resembles morphine.† The cephaëline can be thrown down from the alkaline liquid by adding chloride of ammonium, when it separates in crystals, or the soda may be neutralized with acid, ammonia added, and the cephaëline removed by agitation with ether. These processes must be repeated several times to insure a perfect separation of the alkaloids. We extracted the alkaloids from a sample of Carthagena ipecacuanha root, kindly sent us by Mr. W. Chattaway, by moistening the ground root with ammonia and percolating with amylic alcohol, this process being recommended by Mr. A. J. Cownley. The alkaloids extracted had the following percentage composition :

Emetine	... ...	50·9 per cent.
Cephaëline	... ...	43·8 ,,
Psychotrine	... ...	5·3 ,,

The alkaloids isolated by the foregoing method were assumed to be pure, but we believe this was not strictly the case.

The alkaloids were isolated by the same process from a sample of Brazilian (Rio) ipecacuanha root, but the relative proportions were not ascertained.

We have also isolated the mixed alkaloids from several samples of liquid extract of ipecacuanha (British Pharmacopœia), and separated the alkaloids from one of these, without determining the amount, for the examination of their colour-reactions.

The process employed for the extraction of the mixed alkaloids was to evaporate the alcohol, dilute the residue with water, acidulate the liquid with hydrochloric acid, and agitate with amylic alcohol. This treatment would remove any glucosides present and other bodies of a non-alkaloidal nature. The aqueous liquid was then treated with sodium bicarbonate, and agitated with amylic alcohol to dissolve the alkaloids. This process gives a fairly constant product, but in order to insure the absence of interfering substances in some cases the sample was diluted with water and treated with lead acetate to precipitate various substances, the alkaloids being subsequently extracted from the filtrate after removing the excess of lead.

* It is possible that psychotrine is a decomposition product. It is known that ipecacuanha extracts differ much in the proportion of alkaloid insoluble in ether.

† The solubility of cephaëline in caustic alkalies suggests the presence in its molecule of a hydroxyl-group, which is known to exist in the morphine molecule.

The colour-tests were made in each case by taking up the alkaloidal solution in a pipette and allowing it to fall, drop by drop, on to the concave side of a cover of a porcelain crucible placed on a flask full of boiling water. To the spot of alkaloidal residue thus obtained a drop of reagent was added by means of a glass rod, and the mixture cautiously stirred. Under these circumstances the alkaloids from extract of ipecacuanha gave, when treated with ferric chloride, a blue coloration changing to green, as against a greenish blue from a residue of opium alkaloids obtained in the same manner. Fröhde's reagent gave colours varying from bluish purple to violet. The colour closely resembled that given by mixed opium alkaloids, but was not so bright as that yielded by pure morphine. Starch and iodic acid gave with some extracts of ipecacuanha an immediate blue coloration, but a negative or tardy result was obtained in other cases. Opium alkaloids, of course, gave an immediate blue. All preparations of opium and ipecacuanha immediately reduced a mixture of ferric chloride and potassium ferricyanide with production of Prussian blue.

The isolated and purified alkaloids of ipecacuanha in some instances gave reactions less striking than those obtained with the mixed alkaloidal residue. Psychotrine appears to be the constituent of ipecacuanha alkaloids which gives colour-reactions with ferric chloride and iodic acid, but we believe this to have been due, in part at least, to the presence of another body which could be separated by acetate of lead. At any rate, by treating the extract of ipecacuanha with lead acetate, and decomposing the precipitate with sulphuretted hydrogen in the usual way, we obtained a substance which was removed by amylic alcohol from acidulated solutions, and yielded an extract which immediately reduced iodic acid and mixed ferric chloride and ferricyanide. On rendering the acidulated liquid left from the amylic alcohol treatment alkaline with sodium bicarbonate, and again shaking with amylic alcohol, a substance was obtained which gave a bluish-green coloration with ferric chloride, a dirty purple with Fröhde's reagent, and an immediate blue with mixed ferric chloride and ferricyanide. These reactions appear to point to the presence of a body apparently alkaloidal in nature, which is precipitated by lead acetate.

An objection which applies to many of the colour reactions of alkaloids is that the colours obtained are really due to reduction-products of the reagents employed. Thus, for instance, almost any reducing agent will produce Prussian blue from a mixture of ferric chloride and potassium ferricyanide. If the substance has a somewhat strong reducing power, it will liberate iodine from iodic acid, and thus will give a blue coloration with starch. Similarly, the various shades of green, blue, and purple, obtained with Fröhde's reagent, are doubtless due to the formation of lower oxides of molybdenum, though the actual colours are sometimes curiously constant for the same alkaloid.

Quite recently there has been published in the *Archiv. der Pharmacie* (September 10, 1902), by G. Frerichs and N. de Fuentis Tapis, a paper on "The Assay of Ipecacuanha." These observers generally confirm the observations of Paul and Cownley. The authors state that both emetine and cephaëline dissolve in Fröhde's reagent almost without coloration, but that the addition of a trace of sodium chloride or hydrochloric acid immediately produces an intense indigo-blue

coloration in the case of cephaëline, but not with emetine. Frerichs and Tapis appear to have actually dissolved the alkaloids in excess of Fröhde's reagent, which is a very objectionable method, and one which results in the destruction of all delicacy of the reaction.

According to our experience, when a drop of Fröhde's reagent is applied to an alkaloidal residue on porcelain, emetine gives, as above stated, a dirty-green colour, but this is changed by addition of a minute quantity of hydrochloric acid to a fine grass-green. Cephaëline gives a purple coloration instantly, changed by the addition of hydrochloric acid to a magnificent Prussian blue. Psychotrine gives a dull purple with Fröhde's reagent, changed by hydrochloric acid to a pale green. Opium alkaloids, when similarly treated, give the characteristic purple on addition of Fröhde's reagent, but this colour fades on the addition of hydrochloric acid. The mixed alkaloids from ipecacuanha give the intense blue reaction of cephaëline with great distinctness on addition of hydrochloric acid. This is a readily applied and highly characteristic colour reaction for mixed ipecacuanha alkaloids, and one which distinguishes them quite sharply from opium alkaloids.

Of course, extraction of the recently precipitated ipecacuanha alkaloids with chloroform affords a method of separating them from morphine when there is sufficient material to work on.

Colour-reactions of the Alkaloidal Residues from Amylic Alcohol Extractions.

Reagent.	Ipecacuanha Extract, No. 1.	Ipecacuanha Extract, No. 2.		Ipecacuanha Extract, No. 3.		Opium Alkaloids.
	Direct Process.	Direct Process.	Lead Acetate Process.	Direct Process.	Lead Acetate Process.	
Ferric chloride.	Blue, changing to green.	Blue, changing to green.	Indefinite.	Blue, changing to green.	Blue, changing to green.	Greenish-blue.
Fröhde's reagent.	Bluish-purple.	Purple.	Pink, changing to blue and green.	Violet-blue, changing to dirty pink.	Violet-blue, changing to dirty pink.	Purple.
Fröhde's reagent and hydrochloric acid.	Deep blue.	Deep blue.	Deep blue.	Deep blue.	Deep blue.	Purple (fading).
Starch and iodic acid.	Immediate blue.	Blue, changing to green.	Negative.	Negative.	Pink, changing to blue slowly.	Immediate blue.
Ferric chloride and potassium ferricyanide.	Immediate blue.	Immediate blue.	Immediate blue.	Immediate blue.	Immediate blue.	Immediate blue.

COLOUR-REACTIONS OF THE ISOLATED ALKALOIDS OF IPECACUANHA.

Reagent.	Emetine.	Cephaëline.	Psychotrine.
Ferric chloride.	Indefinite.	Bluish-green (B). Indefinite (C).	Pale cherry red (B). Indefinite (C).
Fröhde's reagent.	Dirty green (B). Bluish (C).	Pink, changing to green (B). Reddish-purple (C).	Pale pink (B). Dull purple (C).
Fröhde's reagent and hydrochloric acid.	Grass green.	Prussian blue.	Pale pink, changing to pale green.
Starch and iodic acid.	Negative.	Negative.	Blue.*
Ferric chloride and potassium ferricyanide.	Gradual blue coloration.	Almost immediate blue (B). Immediate blue (C).	Immediate blue.

The foregoing tables show the colour-reactions we have observed with the alkaloids of ipecacuanha, both mixed and separated. For comparison, we give also the reactions of the mixed alkaloids of opium, as extracted from laudanum.

In some respects the separated alkaloids from Brazilian (Rio) ipecacuanha did not give absolutely identical reactions with the parallel alkaloidal preparations from Columbian (Cartagena) ipecacuanha. Where such differences were observed, the source of the alkaloidal residue is indicated by a B or C in parenthesis.

A most valuable means of detecting ipecacuanha alkaloids consists in the production of psychotrine in a crystallized form. Paul and Cownley describe the crystals as well-defined transparent prisms of a pale lemon-yellow colour. As obtained and observed by us under the microscope, psychotrine forms very minute crystals, which appear to belong to the regular system. Many of them appear to be octohedra, and closely resemble microscopic crystals of arsenious oxide. Other crystals present a remarkable resemblance to granules of rice-starch. Crystals of psychotrine for microscopic observation are readily obtained by shaking out an amylic alcohol or chloroform solution of the alkaloid with a little dilute acetic acid. The acid liquid is separated, concentrated if necessary, and placed in a watchglass, or, preferably, on a microscope-slide furnished with a cell. A watch-glass or small beaker is then moistened internally with ammonia, and inverted over the alkaloidal acetate solution. After a time the vapours of ammonia are absorbed, and liberate the alkaloid in characteristic crystals, which are observed under the microscope. There is no occasion to employ pure psychotrine for the purpose, the crystals being readily obtainable from the mixed alkaloids of ipecacuanha.

We desire to express our obligation to Mr. John Evans for his assistance in preparing various specimens of the alkaloids of ipecacuanha in a state of purity.

* The blue coloration with starch and iodic acid was much more strongly marked in the case of the psychotrine isolated from Brazilian root than with that from Columbian root.

THE ANALYSIS OF PREPARATIONS CONTAINING OPIUM.

By Alfred H. Allen and G. E. Scott-Smith.

(*Read at the Meeting, November* 5, 1902.)

Our attention has been frequently called to the analysis of medicines containing certain preparations of opium, especially paregoric and cough mixtures of various kinds. The analysis of such complex mixtures is by no means simple, and we do not claim to have arrived at a complete method of examination. Paregoric elixir, or compound tincture of camphor of the British Pharmacopœia, is a typical preparation of the kind. It was formerly directed to be prepared from 40 grains each of opium and benzoic acid, 30 grains of camphor, and 30 minims of oil of anise, the whole being diluted with proof spirit to 20 fluid ounces. In the last edition of the British Pharmacopœia (1898) the opium is replaced by an equivalent amount of tincture of opium, the composition of the preparation remaining otherwise unchanged. The composition of paregoric of commerce varies considerably. The spirit being the most costly ingredient offers an inducement to reduce the proportion present. This practice is objectionable, since the prescribed proportion of oil of anise cannot be kept in solution in a weak spirit. Sometimes only traces of oil of anise are present, in which case the paregoric remains clear when diluted with three or four times its volume of water. The benzoic acid is sometimes deficient in quantity, and is occasionally wholly absent, even in the case of tinctures purchased from registered pharmacists. The opium is the most important remedial constituent of paregoric elixir, and is apt to be deficient in amount or quality, besides being frequently wholly omitted. The last practice is, of course, due to the fact that preparations of opium cannot be legally sold except by registered pharmacists, and hence a preparation destitute of opium is largely substituted by general shopkeepers for the genuine paregoric or compound tincture of camphor of the Pharmacopœia. So-called "paregoric substitutes" and "paregoric without opium" are extensively prepared and sold by shopkeepers, and are even vended by costermongers in the streets of London. Some of these preparations are made simply by omitting the opium from ordinary paregoric. In one instance within our experience the opium of paregoric elixir was replaced by hyoscyamus. Potassium and ammonium bromides are also used in factitious paregoric.

If a measured quantity (25 c.c.) of paregoric be rendered distinctly alkaline with caustic soda, and evaporated to about 10 c.c., the alcohol and a portion of the camphor and oil of anise will be volatilized, and the amount of alcohol can be deduced with sufficient accuracy from the specific gravity of the distillate. On shaking the residual liquid with ether, the remaining camphor and oil of anise will be extracted. If the ether be separated, and the aqueous liquid acidulated with hydrochloric acid, benzoic acid will in some cases be precipitated; but whether it separates or remains in solution it can be dissolved out by agitating the acidified liquid with ether. On allowing the separated ethereal solution to evaporate spontaneously in a small beaker, the benzoic acid is obtained in a state fit to weigh; but a better and more

rapid plan is to repeatedly agitate the ethereal liquid with water until the washings no longer redden litmus, add a little more water and a few drops of phenolphthalein solution, and titrate the liquid with $\frac{N}{20}$ caustic alkali (preferably baryta-water), which should be added until the aqueous layer acquires a pink colour, not destroyed by agitation with the ether. Each 1 c.c. of $\frac{N}{20}$ alkali required represents 0·0061 gramme of benzoic acid. If 25 c.c. of the tincture has been employed, the number of milligrammes of benzoic acid found, multiplied by 0·35, gives the grains of benzoic acid per pint of the tincture. The meconic acid extracted, together with the benzoic acid, is too small in quantity to affect the result, but its presence may be detected and the amount roughly determined by separating the ethereal layer after the titration is complete, and destroying the pink colour of the aqueous liquid by a drop of dilute hydrochloric acid. On now adding a drop of ferric chloride solution the deep purplish-red coloration characteristic of meconic acid will be produced. Meconic acid is, however, extracted with difficulty and imperfectly by agitating its acidulated aqueous solution with ether. Amylic alcohol is a far better solvent.

The detection of meconic acid in the above manner of course proves the presence of opium in the tincture. When this information alone is sought the paregoric may be diluted in a test-tube with proof-spirit till it is of a light yellow colour, and a drop or two of solution of ferric chloride then added. If opium be present, a more or less deep red coloration will be produced, owing to the formation of iron meconate. By comparing the depth of red colour with that given by a standard tincture a rough indication of the proportion of opium present can be obtained; but the amount of meconic acid in opium is too variable to allow of much stress being placed on the result obtained. It sometimes happens that paregoric is coloured with cochineal or contains a variety of tannin, in which case the coloration with ferric chloride becomes obscured. On cautiously adding hydrochloric acid, drop by drop, the colour produced by iron tannate is destroyed, while that due to the meconate persists till considerably more acid has been added.

The proportion of opium in paregoric is too small to allow of the ordinary method of determining morphine being conveniently used; but fair results, sufficiently accurate for most purposes, may be obtained by volumetric or colorimetric application of the reaction with iodic acid.

In addition to paregoric and its constituents, cough mixtures often contain other ingredients, among which may be mentioned oil of peppermint, squills, senega, horehound, and preparations of ipecacuanha. Oil of peppermint is readily recognised by the taste and odour. Squills are not readily recognised, the most hopeful plan being based upon the isolation and recognition of quillain. Senega is remarkable for the magnificent purple colour which it yields with strong sulphuric acid. The principle which gives the coloration is not extracted by amylic alcohol from either acid or alkaline solutions.

We are indebted to Mr. A. R. Tankard for valuable experiments on these and other points.

Ipecacuanha may be said to be almost a normal constituent of cough mixtures, and is of interest on account of the close resemblance of some of the colour-reactions of its alkaloids to those yielded by opium alkaloids.

A remarkable case in this connection came within our experience a few months since. A cough mixture was sold which was expected to contain opium both as laudanum and as paregoric. The analyst to whom it was submitted reported it to contain morphine in a proportion of 0·12 per cent., or more than ½ grain per ounce. A small portion of the same sample came into our hands, and we commenced its examination in the full expectation of confirming this result. Hence we submitted the mixture to a process adapted for the separation of morphine. For this purpose the sample was diluted with water, acidulated with hydrochloric acid, and the acid liquid treated with ether to remove the essential oils and any glucosides present. The acid liquid separated from the ether was then shaken with amylic alcohol, and that again separated in its turn. It was next neutralized with sodium bicarbonate and shaken with hot amylic alcohol to insure the extraction of any morphine present. The solution thus obtained was washed with water, and small portions evaporated for the application of colour-tests. The alkaloidal residue from the amylic alcohol gave only a doubtful reaction with ferric chloride,* but a very well-developed morphine reaction with Fröhde's reagent (sulphomolybdic acid), and an immediate reduction with iodic acid and a mixture of ferric chloride and ferricyanide of potassium. On the other hand, we could obtain no microscopic crystals of morphine, as may easily be obtained from such preparations containing morphine, by shaking out the amylic alcohol solution with a little dilute acetic acid, placing a few drops of the acetate solution in a watch-glass, or on a celled microscope-slide, and covering it with a watch-glass moistened with strong ammonia, so that the vapour may act on the liquid in the lower glass. After allowing it to stand for half an hour, the liquid is examined under the microscope, when the elongated prisms characteristic of morphine are readily detected. In this case, however, we failed wholly to obtain the long prismatic crystals characteristic of morphine, but obtained crystals of an entirely different kind, some of which were octahedra, closely resembling arsenious oxide, whilst others suggested the appearance of rice-starch.

At this stage our attention was called to the fact that, at an examination in analytical chemistry held last January, the candidates were required to analyse a liquid extract of ipecacuanha. Of the six who examined the extract several reported it to contain morphine. We have personally questioned three of these gentlemen, and find that they estimated the alkaloid by extracting it from an alkaline solution with amylic alcohol, and reported the presence of morphine as the result of several more or less satisfactory colour reactions. One of the candidates has stated in evidence the facts relating to this examination. Of course, by suitable means the ipecacuanha alkaloids could have been differentiated and separated from any morphine present, but the point is that, when a process suitable for isolation and determination of morphine was used, an alkaloidal residue was obtained which simulated morphine so as to be mistaken for that alkaloid. We consequently prepared the mixed alkaloids from several samples of the liquid extract of ipecacuanha by the same method which we had employed for examining the cough mixture in

* This result we attribute to the fact that we worked on a very limited amount of material. Both opium and ipecacuanha alkaloids give the well-known coloration, but the reaction is not nearly so delicate as that with Fröhde's reagent.

question. We found the alkaloidal residue to give a well-marked greenish-blue coloration with neutral ferric chloride; a crimson changing to purple and blue colour with Fröhde's reagent; an immediate blue coloration with iodic acid and starch (with certain samples, but not with all); and an immediate formation of Prussian blue from a mixture of ferric chloride and potassium ferricyanide. The microscopic crystals obtained were, however, exactly similar to those which had been prepared from the cough mixture, and in no way resembled morphine. The crystalline appearance was so characteristic that we had no hesitation in reporting that the cough mixture contained ipecacuanha, and, in view of the colour reactions which extract of ipecacuanha of undoubted purity had given, we could find no evidence of the presence of morphine. We had searched for meconic acid, with wholly negative results, by the method which we had found serviceable in other cases—namely, extraction of the acidified liquid with amylic alcohol, evaporation of the solution, and testing the residue with ferric chloride.

After reporting the presence of ipecacuanha, the person who compounded the cough mixture informed us that he had actually introduced ipecacuanha wine into the cough mixture in question, but had not made any addition of a preparation of opium. It appeared evident, therefore, that the analyst who originally examined the mixture had mistaken the ipecacuanha alkaloids present for morphine.

The case affords an interesting example of the danger of relying on colour-indications for alkaloids without a definite assurance that no interfering body is present. The remarkable manner in which the alkaloids of ipecacuanha similate those of opium in some of their reactions is not generally known, and we have felt it to be of such practical importance as to warrant us in bringing the matter before the Society in the detailed manner we have done.

Discussion.

Mr. William Chattaway stated that Mr. Allen had afforded him the opportunity of examining a portion of the original cough mixture, the composition of which had been such a vexed question, and he was able to confirm the authors' statements in every respect. The cough mixture gave several of the colour-reactions of morphine, but Fröhde's reagent, followed by hydrochloric acid, gave the magnificent Prussian blue coloration which had recently been found so characteristic of cephaëline. He also confirmed the authors' statements with respect to the characteristic crystals from ipecacuanha which they attributed to psychotrine. There was no difficulty in obtaining from the sample of cough mixture in question numerous microscopic crystals of this kind, and they were quite distinct in character from crystals of morphine obtained in an analogous manner.

Mr. Allen said that he was glad to know that his experience in this matter was so fully confirmed by that of Mr. Chattaway. There was no reflection on anyone who had made a mistake in the past when these reactions were not recognised, but care in the future was obviously necessary lest a preparation containing ipecacuanha might be mistaken for one containing the alkaloids of opium, especially as these colour-tests were much less definite and striking with the mixed

alkaloids of opium than with pure morphine. The remarkable reaction for cephaëline with Fröhde's reagent and hydrochloric acid was of great assistance, but it was conceivable that the reaction with Fröhde's reagent might be complicated by the presence of other substances, and he thought that the most definite and characteristic evidence of the presence of ipecacuanha was really that afforded by the production of crystals of psychotrine.

Mr. Moor mentioned that with regard to tinct. camph. co. a standard had been recently issued by the Irish Local Government Board. That standard did not render it necessary to go very deeply into the question, but simply laid down the specific gravity, the alcoholic strength (57 per cent. by volume), and the total solids, which were put at 0·33 per cent. He was inclined to think, however, that that percentage of total solids was somewhat too high, and would like to hear Mr. Allen's opinion on this point.

Mr. Allen said that he was afraid he could not, without reference, give any figure for the total solids in paregoric. It was to be borne in mind, however, that at present this preparation was directed to be made, not with opium, of which the quantity used to be 40 grains per pint, but with an equivalent quantity of tincture of opium. The solids of the paregoric would therefore consist of the soluble matter of the opium from which the tincture was prepared, assuming all the other constituents of the paregoric to be volatile; so that from a knowledge of the average amount of solid matter contained in the laudanum—which, however, was liable to some variation—it might be ascertained by calculation whether the standard was correct.

Mr. Moor said that the quantity of tincture of opium directed to be taken was 60·9 c.c. per litre, and, assuming the average solids of the tincture to be 4 per cent., or a minimum of 3 per cent., the total solids of the paregoric might fall as low as 0·18 per cent., assuming the benzoic acid and oil of anise to be entirely volatile; and, as a matter of fact, he had figures of his own showing as little as 0·2 per cent. in a number of samples which he believed to be quite genuine. He mentioned this because the standard referred to was intended to form the basis of prosecutions; and if that were insisted upon, any sample falling below 0·33 per cent. in total solids would be prosecuted on. He thought, however, that it was not quite correct to place the standard so high.

Mr. R. A. Cripps said that he could quite confirm Mr. Moor's remark that paregoric might be perfectly genuine, and yet contain not more than 0·2 per cent. of total solids. In the examination of cough mixture, it seemed to him that ipecacuanha was almost the first thing to be expected; and with the knowledge that the alkaloids of ipecacuanha were soluble in amylic alcohol, a reasonable course would seem to be to extract first in the alkaline condition with ether, which would remove the cephaëline and emetine and leave the morphine.

Mr. Allen said that psychotrine would also remain undissolved after such a process as that suggested by Mr. Cripps, and psychotrine was the alkaloid which gave the characteristic crystals he had described. Chloroform would be preferable, but he would be very shy of treating a small quantity of such a preparation as a cough mixture with ether or chloroform for the purpose of removing one class of

alkaloids and leaving the other. As a matter of fact, however, his purpose had been rather to describe the way in which the matter had actually been dealt with in the past than to suggest what might be the best way of dealing with it.

Mr. Cripps said that, as far as his experience went, there was no practical difficulty in the treatment with ether for removing the alkaloids other than morphine.

NOTE ON HONEY.

By Henry Leffmann.

Some time ago I was consulted by a manufacturer of table accessories in regard to a sample of honey which had been reported by one of the State chemists as adulterated. The manufacturer did not have any portion of the sample which had been analysed, but brought me some samples which he believed to be from the same lot, and said that the material was from California, and was packed as received, being believed to be pure. Inquiry elicited the fact that the sample purchased by the State's agent had been found to give the following polarimetric results on the ordinary sugar-scale :

Before hydrolysis	9·0
After hydrolysis	3·0

The sample submitted to me gave results of the same import, although not exactly the same figures, namely :

Before hydrolysis	13·5
After hydrolysis	9·5

Dextrorotatory honey is usually looked upon with suspicion. When the sample remains strongly dextrorotatory after hydrolysis, the addition of commercial glucose is assumed.

It is, however, established that pure honey may be dextrorotatory. The following figures are reported on good authority as given by samples of undoubted purity :

Before hydrolysis	8·2	7·2	7·3
After hydrolysis	2·8	3·3	2·6

Polarimetric data are so technical that it is desirable to have some chemical tests for the presence of glucose as such. Most of the data that are herewith presented are principally of value as embodying the application of German methods to American samples, and, as will be seen, confirming the claims of the German chemists.

Commercial glucose is by no means the simple substance that it was supposed to be when first manufactured. It contains, in addition to dextrose, considerable dextrin and maltose, some nitrogenous matter, and a notable amount of unfermentable optically active carbohydrates of uncertain nature, among which is one called gallisin. As strong alcohol precipitates dextrin from glucose, the suggestion to test with this would naturally occur; but although honey is largely invert-sugar, it

frequently contains considerable amounts of dextrinous matter that precipitates with strong ethyl-alcohol. Bechmann suggested the use of methyl alcohol, and tests I have recently made have shown the value of this method. The refined methyl alcohol, termed "Columbian spirit," is preferable. Two methods are employed. The simplest consists in mixing the honey with an equal weight of water and then adding a large volume of the alcohol. The quantities used have been 8 grammes of honey, 8 c.c. of water, and 100 c.c. of alcohol. The characteristic of glucose is to give a sticky precipitate which may become somewhat granular, and which adheres to the wall of the vessel. Pure honey gives a light flocculent precipitate, which does not adhere to the glass.

For the second test a solution of 20 grammes of the honey in 100 c.c. of water is prepared. Five c.c. of this solution are mixed with 3 c.c. of a 2 per cent. solution of barium hydroxide and 17 c.c. of methyl alcohol, and the mixture shaken with as little contact with air as possible. Pure honey gives but little precipitate, but in the presence of glucose or glucose syrup a considerable precipitate is formed.

These tests applied to the suspected honey and to samples believed to be pure, and also to mixtures of such samples with commercial glucose, gave very characteristic results. The barium method is unsatisfactory on account of the action of the air, which cannot be easily avoided. An attempt to substitute a solution of barium carbonate in water containing carbonic acid did not succeed. Apparently the direct action of the hydroxide upon the carbohydrates is needed.

It seemed probable that useful information might be obtained by comparing the condition of solutions before and after fermentation, including the polarimetric data, especially as applied in the examination of wine. The following experiments were made: Three solutions were prepared by dissolving 25 grammes of material in water made up to 250 c.c. The polarimetric reading of each was taken, and the solutions were then fermented at room temperature (over 20° C.) for six days, and again examined. A few drops of a solution of disodium hydrogen phosphate and ammonium carbonate had been added to each. The fermentation was quite active for several days. The results were as follows:

	Polarimetric Reading.		Fermented Solution.	
	Before Fermentation.	After Fermentation.	Specific Gravity.	Solids per Cent.
Pure honey	−6·0	3·0	1,003	1·6
Commercial glucose	65·0	43·7	1,014	4·2
One-half honey, one-half glucose ...	28·6	22·5	1,009	2·8

The above data are too few to form a basis for argument, but they tend to show that the fermentation method may aid in elucidating the nature of sample under dispute.

Some experiments were made with a view of determining the polarimetric character of solutions of samples in methyl alcohol. Crude methyl alcohol was used. In each case 8 grammes of the sample were treated with 100 c.c. of methyl alcohol, well mixed, and allowed to stand for more than a day. The clear liquid was then

decanted and the reading taken. All samples were examined at the same temperature. The results were:

Sample.	Precipitate with Methyl Alcohol.	Polarimetric Reading.
Pure honey	Slight, flocculent	1·0
Suspected honey	Marked, granular	22·0
Pure honey (glucose equal parts) ...	Marked, very sticky	31·0
Glucose	Marked, sticky	10·5

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOODS AND DRUGS ANALYSIS.

The Detection of Added Water in Milk by Means of the Nitrate Test. M. Siegfeld. (*Molkerei-Ztg. Hildesheim*, 1902. xvi., 161, 162; through *Zeit. für Untersuch. der Nahr. und Genussmittel*, 1902, v., [18], 867.)—The author has compared the various reactions for detecting the presence of nitrates in milk, and finds that the formaldehyde test is as sensitive as the diphenylamine reaction. The former test should be made by allowing the milk, to which a drop of formaldehyde has been added, to run on to the surface of pure sulphuric acid. If nitrates be present a violet ring is formed at the junction of the two liquids. It is not advisable to mix the acid and milk, as milk containing no nitrates gives a reddish-violet coloration, which may be mistaken for the nitrate reaction.

(Note by Abstractor.—Should the sulphuric acid contain a trace of iron, a blue ring is always obtained with milk containing formaldehyde, whether nitrates be present or absent.) W. P. S.

Method for the Estimation of Metallic Impurities in Condensed Milk. J. W. Abbott. (*Bull. Massachusetts Health Dept.*, 1900, 37; through *Zeit. für Untersuch. der Nahr. und Genussmittel*, 1902, v. [18], 866.)—The condensed milk is mixed in a china basin with a third of its weight of sulphuric acid, and subjected to an electric current of 1 to 4 ampères. After fifteen minutes' treatment the whole mass becomes thorougly charred. The incineration is then readily completed over a free flame, and the ash may be employed for the estimation (electrolytical) of the metallic impurities which may be present. W. P. S.

Determination of the Casein precipitated by Rennet. L. Lindet. (*Ann. de Chim. anal.*, 1902, vii., 361-363.)—According to the author's experience, the methods of determining casein in milk are not reliable, for the precipitation is

invariably incomplete; and there is therefore no exact method of determining the value of milk for the purpose of cheese manufacture. The quantity of casein precipitated by rennet under definite conditions can, however, be rapidly calculated from the depression in the specific gravity of the separated milk after the addition of the enzyme. From the average results of his experiments the author finds that on the average the precipitation of each 3·5 grammes of casein per litre lowers the specific gravity of the whey by 1 degree. Hence the difference between the specific gravity of the separated milk and of the whey multiplied by 3·5 gives the amount of casein per litre precipitated by the rennet.

Instead of separating the fat from the milk the specific gravity of the fat-free milk can be calculated by means of the formula—

$$100\ D = a \times 940 + (100 - a)D',$$

$$\text{or } D' = \frac{100\ D - a \times 940}{100 - a},$$

where D represents the specific gravity of the whole milk expressed in grammes, D′ that of the separated milk, 940 that of the butter-fat in the milk, and a the percentage of butter-fat. C. A. M.

Separation and Estimation of Cholesterol in Butter Fat. A. Kirsten. (*Zeit. für Untersuch. der Nahr. und Genussmittel*, 1902, v. [18], 849-856.)—Ten grammes of the melted fat are heated on a boiling-water bath under a reflux condenser for fifteen minutes with 20 c.c. of alcoholic potash, made as follows: 2 volumes of concentrated potassium hydroxide solution—1,000 grammes per litre—1 volume of water, and 7 volumes of absolute alcohol. Forty c.c. of water are then added, and, after cooling, 50 c.c. of ether. The contents of the flask in which the saponification has taken place are transferred to a separating funnel, and the flask is rinsed out with 50 c.c. of ether several times. The funnel is then shaken vigorously for one minute, and allowed to stand, the contents quickly separate. The ethereal layer is removed, and the soap solution shaken out five times with ether, using 50 c.c. of the latter each time. Should an emulsion form, a little alcohol may be added. The ether is distilled off from the combined extracts, and the alcohol remaining in the residue is removed by evaporation on the water-bath. The residue is then again heated with 10 c.c. of alcoholic potash, to saponify traces of fat which it still contains, diluted with 30 c.c. of water, and shaken out with 100 c.c. of ether. After drawing off the soap solution, the ethereal layer is washed three times with 10 c.c. of 5 per cent. potassium hydroxide solution and twice with 10 c.c. of water. These washings are not added to the soap solution. The ether is filtered into a weighed flask. After once more shaking the soap solution with 100 c.c. of ether, this second ethereal portion is also filtered into the weighed flask, and the filter washed with a little ether. The ether is then distilled off, and the residue of cholesterol is dried and weighed. The average amount of cholesterol found by the author in butter fat was about 0·46 per cent. At the commencement of lactation the quantity was 0·37 per cent., increasing at the end of the lactation period to 0·50 per cent. W. P. S.

Detection of "Vinegar Essence" in Fermentation Vinegar. F. Rothenbach. (*Deutsche Essigindustrie*, 1903, vi., 49, 50 and 59-64; through *Zeit. für Untersuch. der Nahr. und Genussmittel*, 1902, v., [17], 817-819.)—The following methods are given:

1. Fifty c.c. of the vinegar are shaken in a separating-funnel with 20 to 30 c.c. of pure, alcohol-free chloroform. An emulsion is avoided by adding water and well cooling the mixture. The chloroform layer is filtered through a dry filter and again cooled, when a turbidity forms. On adding 2 to 3 c.c. of a mixture of 10 parts of concentrated sulphuric acid and 11 parts of fuming nitric acid, a dark-red zone appears between the two liquids should fermentation vinegar be present. By carefully shaking the mixture the whole chloroform layer becomes red-coloured, the acid layer remaining colourless. The red colour is very stable. Vinegar essence gives no coloured zone.

2. One c.c. of the sample, 0·1 c.c. of $\frac{N}{10}$ iodine solution, and 0·2 c.c. of concentrated sulphuric acid are mixed in a test-tube and cooled. Concentrated vinegar essence gives a clear, dark-red coloration, and water containing 1 per cent. of essence a clear yellow colour. Pure fermentation vinegar yields a dark-red coloration; the fluid soon becomes turbid and opaque, and a greenish layer forms on the surface. Dilute fermentation vinegar gives a dark-red colour, soon becoming turbid. Mixtures of 100 c.c. of 10 per cent. fermentation vinegar with 20, 10, and 5 c.c. of vinegar essence give clear red colours; with less than 2·5 c.c. the mixture is still red, but soon becomes turbid. When fermentation vinegar is distilled the distillate yields a clear, bright-yellow colour, whilst the residue gives a turbid red coloration. Vinegar essence which has been allowed to stand in contact with vinegar shavings for a considerable time shows, when tested by this method, a dark-red colour which remains clear.

3. The vinegar to be tested is diluted down to 1 per cent. Twenty c.c. of this dilute solution are placed in a wash-bottle, which is connected to a second wash-bottle containing 5 c.c. of sulphuric acid (1 : 3) and 19 c.c. of $\frac{N}{100}$ permanganate solution. A strong regular current of air, which has first been passed through an alkaline solution of permanganate, is drawn through the vinegar and permanganate solutions for two minutes. The wash-bottle containing the latter is then detached and placed for five minutes on a boiling-water bath. Ten c.c. of $\frac{N}{100}$ oxalic acid solution are then added, and the titration completed with $\frac{N}{100}$ permanganate. A blank test should be made, using 20 c.c of water. Vinegar essence destroys practically no permanganate. Fermentation vinegar, according to the amount of alcohol, etc., it contains, uses at least 1 c.c. of the permanganate. An attempt is made to render this test quantitative, but without satisfactory results. W. P. S.

The Estimation of Caffeine. J. Katz. (*Zeits. f. angew. Chem.*, 1902, xxxix., 1013.)—The method is a modification of that of Beitter. Of the sample, 10 grammes are taken and shaken for half an hour with 200 grammes of chloroform and 0·5 gramme of ammonia. After settling, the chloroform solution is filtered through a Sander

filter in order to obtain 150 grammes of clear water-free filtrate. The chloroform is distilled off entirely, and the residue is dissolved in 5 c.c. of ether, then 20 c.c. of 0·5 per cent. hydrochloric acid are added, and the ether is boiled away. When cold, the aqueous liquid is filtered, the flask and filter being washed several times with 0·5 per cent. hydrochloric acid, and the acid filtrate is shaken out four times with chloroform, using 20 c.c. each time. The chloroform solution is filtered if necessary, and the solvent is distilled off.

The above method is used for coffee-beans, black tea, guarana, and kola-nuts. In the case of Paraguay tea, the material is treated with chloroform and ammonia as already described, the solution evaporated, and the residue dissolved in ether. Water is then added, the ether is boiled away, and 2 c.c. of water are introduced, having 5 per cent. of lead hydroxide in suspension. The liquid is heated for ten minutes on the water-bath, then a few decigrammes of calcined magnesia are added, and the liquid after cooling is filtered. The filtrate, which should be clear and only slightly coloured, is extracted in a "perforator" for two hours or shaken out with chloroform, and the extract is evaporated down. A. M.

A Test for Phenacetin. F. H. Alcock and W. Wilkins. (*Pharm. Journ.*, 1902, 258.)—A purple coloration is obtained on strongly heating 0·01 gramme of phenacetin with 5 c.c. of pure sulphuric acid. On pouring the solution, when nearly cold, into a large bulk of water and adding ammonium hydroxide, a deep purple-coloured solution results. Should any charring have taken place, the solution must be filtered before making ammoniacal. Phenazone, sulphonal, and acetanilide give entirely different results. W. P. S.

TOXICOLOGICAL ANALYSIS.

The Forensic Value of "Florence's Crystals." N. Bocarius. (*Vierteljahrschr. gericht. Med. u. öffentl. Sanitätsw.*, 1901, xxi., 255-266; through *Zeit. für Untersuch. der Nahr. und Genussmittel*, 1902, v., 942, 943.)—Florence's crystals (see ANALYST, xxiii., 320) are not only obtained with human seminal fluid, but also with that of animals as well as with other animal and vegetable substances, such as extracts from the liver, the blood of common insects, caterpillars, etc. The crystals are always obtained even from the smallest trace of human semen, whether it be wet or dry, fresh or decomposed, except when the semen is coloured emerald-green or orange by micro-organisms. Animal seminal fluid and bodies not of a spermatic nature react more feebly with the iodine (Florence's) solution than does human semen.

The crystals may be obtained with various iodine solutions, but the reagent must contain excess of iodine. The age of the seminal traces does not influence the reaction, nor does a high temperature or decomposition, but the presence of large quantities of blood, and other animal fluids, interferes with the formation of the crystals; an excess of liquid—water or reagent—as well as an excess of potassium iodide in the latter, should be avoided. The form of the crystals is altered according

to the reagent used, and by other causes. The actual substance which forms the crystals in the presence of iodine exists not only in seminal fluids, but also in many organic bodies. Florence's reagent is, therefore, only a selective test. W. P. S.

The Nature of the Substance which causes the Formation of "Florence's Crystals." **N. Bocarius.** (*Zeit. physiol. Chem.*, 1902, xxxiv., 339-346; through *Zeit. für Untersuch. der Nahr. und Genussmittel*, 1902, v., 943.)—To ascertain the nature of these crystals the author carried out a number of experiments with the seminal fluids of men and horses, the livers of oxen, and human brains and livers. These were first treated with 2 per cent. formaldehyde solution, and by acting on the extracts of the various objects with a solution of iodine in sodium iodide large quantities of crystals were obtained. The latter keep well if allowed to stand in the iodine solution. After decomposing the crystals with silver hydroxide, the products were treated with alcohol, and converted into platinum chloride salts. On analysis the latter were found to the following percentages of platinum: human semen, 31·65; animal semen, 31·51; human liver, 31·82; ox liver, 31·53; and human brain, 31·61. The average of these figures is 31·62 per cent., whilst choline-platinum chloride gives 31·64, and semen-platinum chloride 38·21 per cent. of platinum. The author comes to the conclusion that choline must be considered as the substance which forms the crystals with the iodine solution. (See ANALYST, xxiii., 320.) W. P. S.

Determination of Mercury in Toxicological Work. **C. Pierpaoli.** (*Boll. Chim. Farm.*, 1902, xli., 561; through *Chem. Zeit. Rep.*, 1902, 265.)—According to the author, the partial loss of mercury which occurs when animal substances have been destroyed by the Fresenius-Babo process is due to the following causes: In the precipitate produced by sulphuretted hydrogen the mercuric sulphide is accompanied by organic matter containing chlorine, from which it cannot be freed by washing with water; on the contrary, by excessive washing part of the sulphide passes into the colloidal modification, and is therefore lost. When the dry impure sulphide comes to be treated with nitric, and afterwards with sulphuric, acid, in order to complete the destruction of organic matter, part of the mercury is converted into chloride by the chlorine present, and volatilized when the sulphuric acid is warmed. As a matter of fact, Pierpaoli has found that if mercuric chloride is heated with sulphuric acid to a temperature not exceeding 170° C. the salt is not decomposed, but volatilizes unchanged. F. H. L.

ORGANIC ANALYSIS.

The Composition of Human Fat. **H. Jaeckle.** (*Zeit. physiol. Chem.*, 1902, xxxvi., 51; *Chem. Rev. Fett-u. Harz-Ind.*, 1902, ix., 232.)—According to the results of the author's analyses, normal adult human fat consists in the main of glycerides of oleic, palmitic, and stearic acids, with traces of lower fatty acids. The fat of a

child during the first few months shows pronounced differences from the adult fat, being characterized by a high proportion of lower fatty acids and a small proportion of oleic acid. It was not found possible to trace any influence of the food of different individuals upon the composition of the fat. In pathological cases the fat may undergo very great changes (*cf.* ANALYST, xxi., 171). C. A. M.

The Molecular Equivalent of Insoluble Fatty Acids. M. Tortelli and A. Pergami. (*Chem. Rev. Fett-u. Harz-Ind.*, 1902, ix., 182-184, 204, 205.)—The ordinary process of determining the molecular equivalent of fatty acids consists of dissolving a weighed quantity in alcohol and titrating the solution with $\frac{N}{10}$ alkali. It has usually been assumed that the acid value thus found is identical with the saponification value. The authors, however, point out that there is a great discrepancy between the recorded molecular equivalents of the fatty acids of different oils and their known composition. The cause of this source of error, which is clearly shown in the following examples, is to be attributed to the presence of lactones:

Oil or Fat.	Oil or Fat.		Insoluble Fatty Acids.		Mean Molecular Equivalent of Fatty Acids.		Difference.
	Acid Value.	Saponification Value.	Acid Value.	Saponification Value.	Calculated from Acid Value.	Calculated from Saponification Value.	V.-VI.
	I.	II.	III.	IV.	V.	VI.	
Almond oil:							
$2\frac{1}{2}$ years old	1·50	193·6	196·0	202·2	286·2	277·5	8·7
Fresh, cold-drawn ...	0·70	193·6	195·8	203·2	286·5	278·3	8·2
Cotton-seed oil:							
$2\frac{1}{2}$ years old	0·19	195·2	194·3	204·5	288·7	274·3	14·4
Fresh, cold-drawn ...	3·80	198·3	200·9	203·1	279·2	276·2	3·0
Colza oil:							
2 years old	3·7	172·2	178·3	182·5	314·6	307·4	7·2
Fresh, cold-drawn ...	0·55	174·9	176·6	181·2	317·7	309·6	8·1
Castor oil:							
$2\frac{1}{2}$ years old	3·29	—	183·1	189·0	306·4	296·7	9·7
Fresh, cold-drawn ...	1·9	181·3	187·0	191·1	300·0	294·3	5·7
Linseed oil:							
3 years old	2·70	192·6	191·5	205·4	292·8	273·2	19·6
Fresh, cold-drawn ...	0·30	189·8	194·6	201·8	288·2	277·9	10·3
Ox tallow:							
2 years old	4·3	199·6	205·4	207·1	273·1	270·8	2·3
Fresh, rendered by heat	1·4	200·0	205·7	209·9	272·7	267·2	5·5
Lard:							
2 years old	0·2	196·8	203·4	203·1	275·8	276·2	0·4
Fresh, rendered by heat	5·9	196·8	205·0	204·8	273·4	273·9	0·5
Palmitic acid	—	—	202·7	218·7	276·5	256·4	20·1
Stearic acid	—	—	198·7	198·9	282·3	282·0	0·3
Oleic acid, from olive oil...	—	—	199·5	201·4	281·2	278·5	2·7

To obviate this error, the authors hydrate the lactones by boiling the fatty acids with an excess of alkali and titrate the excess, just as in the determination of the saponification value by Koettstorfer's method. C. A. M.

Some Colour Reactions of Fatty Oils. **H. Kreis.** (*Chem. Zeit.*, 1902, xxvi., 1014.)—This is essentially a continuation of the author's previous article (ANALYST, xxvii., 330). It is known that Bishop's reaction—a green colour on shaking with 1·19 hydrochloric acid—only occurs with such sesamé oils as have been bleached by insolation or otherwise. It is also known that, if a sesamé oil that does not give Bishop's reaction is mixed with some other insolated or bleached oil, the whole gives a strong green or blue colour when treated similarly. Moreover, a list of oils was contained in the former paper which no longer yielded the Bellier test, but which behaved with sesamé as above. For convenience this reaction may be termed the Bishop-Kreis test. It is now found that oils which yield the Bishop-Kreis reaction also give colours with resorcinol and with phloroglucinol, the test being preferably made by mixing 2 c.c. of 1·19 HCl with 2 c.c. of oil and 2 c.c. of a cold saturated solution of resorcinol in benzene, or of a solution of 1 part of phloroglucinol in 1,000 parts of ether. After a short time—or rapidly on the water-bath—fine violet or red tints appear in the acid, which are not affected by the addition of water. Hitherto this reaction has been observed with olive, arachis, sesamé, cotton, poppy, and walnut oils, also with two samples of butter which had become tallowy. A sample of arachis, which gave the Bellier test powerfully, but failed to yield the Bishop-Kreis test, was exposed to sunlight for eight (September) days; it then failed to give the Bellier, but gave a notable green with the Bishop-Kreis, and also yielded bright colours with resorcinol or phloroglucinol and HCl. The acid value (4·4) was not affected by the insolation.

F. H. L.

Detection of Sesamé Oil in Earth-Nut Oil. **J. Schnell.** (*Zeit. für Untersuch. der Nahr. und Genussmittel*, 1902, v., 961-963.)—Soltsien's reaction is recommended, the test being carried out as follows: The oil is mixed with an equal volume of stannous chloride (German Pharmacopœia strength) and shaken vigorously, but not repeatedly. The test-tube containing the mixture is then placed in boiling water until separation takes place. A red coloration appears should sesamé oil be present, whilst pure earth-nut oil gives no coloration. Less than 1 per cent. of sesamé oil can be detected, but attention is drawn to the fact that sometimes the same press is used for sesamé seeds as for earth-nuts, and the oil from the latter may, for this reason, show indications of the presence of traces of sesamé oil.

It may be mentioned that the stannous chloride reaction is particularly applicable to butters and margarines artificially coloured with coal-tar dyes. The colours are reduced and rendered colourless when the test is applied, and their previous extraction with dilute hydrochloric acid is unnecessary.

Several samples of earth-nut oil from West Africa examined by the author gave iodine numbers as low as 84·4 to 85·7. Others, from the East Indies, gave higher

numbers than usual—from 89·7 to 95·0. As these numbers are higher than that for pure olein, other unsaturated acids besides oleic are probably present in these samples. W. P. S.

Halphen's Reaction for Cotton-Seed Oil. B. Sjollema and J. E. Tulleken. (*Zeit. für Untersuch. der Nahr. und Genussmittel*, 1902, v., 914-916.)—Butter obtained from cows which have been fed on cotton-seed meal gives with Halphen's test exactly the same coloration as does cotton-seed oil itself. On spectroscopically examining the colour produced in this test the authors found that, at a low temperature (55° C.), cotton-seed oil yielded an absorption band in the yellow with a maximum at λ 550, a thicker layer showing a maximum more to the left at λ 570. By long heating or by employing a higher temperature a second band was produced at λ 490, which gradually increased in intensity. A thicker layer, without dilution, caused the right half of the spectrum to become so dark that observations were rendered impossible. On considerably diluting with amyl alcohol, the band had a distinct maximum at λ 490, but the band in the yellow had lost much of its intensity. W. P. S.

Notes on Some Essential Oils. (*Schimmel and Co.'s Half-Yearly Report*, October, 1902; through *Chem. Zeit.*, 1902, xxvi., 1005.)

Camphor Oils.—Methods of manufacture having been somewhat altered, these oils are rather different from what they formerly were. The light oil has a yellowish colour and a specific gravity of about 0·900; the dark oil is green, and its density is 0·930. A third product is now prepared, viscid, and of a fine blue colour; it is used in porcelain painting. It boils between 280° and 300° C., has a specific gravity of 0·95 to 0·96 at 15° C., and is dextrorotatory, one sample showing a value of +32° 55′. Apparently it consists for the most part of an alcoholic body, for on treatment with acetic anhydride it yields a considerable saponification value. In spite of its colour, it does not tint soap, and it is therefore employed as a fixing agent for ordinary soap perfumes. Little is yet known of the constituents of camphor oil which are soluble in alkali. On distillation aldehydes and acids are obtained, but the latter have not been identified. A comparatively small quantity of eugenol is present in the higher fractions. In the highest acid fractions eugenol and carvacrol have been found; the latter boils at 86° to 88° C. at a pressure of 2 millimetres. Apparently a second product, boiling at 94° to 99° (3 millimetres), is also present. About 3 per cent. of the portion soluble in alkali dissolves in dilute sodium carbonate; it consists of a mixture of the carboxylic acids of the fatty series, caprylic acid being predominant. Through its easily soluble calcium salt a liquid acid boiling at 114° to 115° (4 millimetres) has been recovered from the crude acids; analysis of its silver compound leads to the formula $C_9H_{16}O_2$; it probably belongs to the oleic series.

Oil of Bergamot.—S. Gulli has published an account of this oil. It is only distilled between February and April, when the trees are pruned and stripped; the yield is quite small, 100 kilos of leaves giving but 150 grammes of oil; probably not more than 20 or 25 kilos are made in any year. The pure oil has a specific gravity of about 0·870 to 0·873, its rotatory power is +25° to +26°, and it is soluble in an equal

volume of 90 per cent. alcohol. It contains about 32 to 34 per cent. of esters reckoned as linalyl acetate, part being the methyl ester of anthranilic acid.

Oil of Lemon.—Although a large number of bodies have been identified in this oil—*d*-limonene, cymol (?), phellandrene, citral, citronellal, geranyl acetate, a sesquiterpene, octyl- and nonyl-aldehyde, and pinene—it is not possible by artificially mixing these substances together to produce a useful lemon oil. This is due to the fact that several important odour-giving constituents have been overlooked. Recently in Schimmel's laboratory methylheptenone has been obtained from the distillate of lemon oil, and terpineol (melting at 35°, Δ^1-Terpene-8-ol) recovered from the residue.

Oil of Lemon-grass.—Owing to the lack of prosperity in the sugar industry of the West Indies, it would seem that attempts are being made to cultivate the lemon-grasses in those islands. The superintendent of the botanical gardens in Trinidad has published an account of some investigations made in the Government laboratory at Antigua on this subject. The oil of *Andropogon Nardus var.* has been found to have a specific gravity of 0·9084 at $\frac{15{\cdot}5^\circ}{15{\cdot}5^\circ}$; a specific rotatory power of + 0° 1′; an aldehyde content of 15·5 per cent.; saponification value, 23; and saponification value after acetylization, 168·6, corresponding with about 53 per cent. of total alcohols. The oil of *Andropogon Schœnanthus* gave: Specific gravity, 0·9315 at $\frac{15{\cdot}5^\circ}{15{\cdot}5^\circ}$; rotatory power, +3°; aldehyde content, 48·2 per cent.; saponification value, 31·1; after acetylization, 69·6, corresponding with 20·2 per cent. of $C_{10}H_{18}O$. Apart from its slight dextrorotatory power, the former oil resembles Ceylon citronella oil, for it is not soluble in 10 volumes of 70 per cent. alcohol, but dissolves in the same volume of 80 per cent. spirit; but the latter has very different properties from Palmarosa oil, with which it appears to be botanically identical. Palmarosa oil, however, is not a lemon-grass oil, for it contains too little aldehydes, assuming, that is, that its aldehyde is really citral.

Liquid Oil of Musk.—Ordinary oil of musk is solid at ordinary temperatures, owing most probably to the presence of palmitic acid. This being inconvenient in practice, Schimmel and Co. have removed the solid constituent, and have introduced a liquid oil. The new material remains fluid at all temperatures, and is equivalent to six times its quantity of the usual product. The liquid oil has a specific gravity of 0·909, a rotatory power of +1° 10′, an acid value of 2·4, an ester number of 180·5, and it is soluble in 5 to 6 volumes of 80 per cent. alcohol.

Oil of Neroli.—A recent investigation has shown that oil of neroli contains, besides the constituents hitherto recognised, the following bodies: *l*-pinene, *l*-camphene (?), dipentene, decyl-aldehyde (?), an alcohol $C_{10}H_{18}O$ (probably *l*-linalool), phenylethyl alcohol (free, or as ester), *d*-terpineol (melting-point, 35°), phenylacetic acid, and benzoic acid.

Oil of Petit-grain.—A specimen of genuine oil from Paraguay has been found to have a specific gravity of 0·8912, and to contain furfurol, *l*-pinene (?), *l*-camphene (?), dipentene, an alcohol $C_{10}H_{18}O$ (*l*-linalool), *d*-terpineol (melting-point, 35°), geraniol, geranyl acetate, and traces of a basic substance.

Italian Oil of Peppermint.—A sample of this oil, distilled in Piedmont, had

a pale greenish-yellow colour and an odour somewhat resembling pennyroyal. Its constants were: specific gravity at 15°, 0·9122; rotatory power, −16° 21′; solubility, 7 volumes of 70 per cent., 1·1 volume of 80 per cent. alcohol; total menthol content, 52·5 per cent., 7·89 per cent. thereof existing as esters. The sample, accordingly, did not solidify in a freezing mixture. The menthone, on the contrary, was high—viz., 22 per cent. With larger quantities of alcohol than those mentioned the oil gave a transient opalescence.

Oil of Aniseed.—Tardy has recently investigated this oil. After removing the bulk of the anethol, the sample had a rotatory power of −3° 15′. Besides the constituents already known—pinene, phellandrene, methylchavicol, the ethyl ester of quinol, anisaldehyde, and anisic acid—Tardy found anisic ketone, a sesquiterpene having a lævorotatory power of 5°, and a body melting at 212°, which is probably identical with the substance melting at 213° C. isolated from oil of fennel. Tardy also considers that oil of aniseed contains terpineol, but beyond quoting the boiling-point of 216° to 218° he gives no proof of its existence.

Oil of Bystropogon Origanifolius.—This is a new material introduced by the firm of L. Rigal in Teneriffe. It is prepared from a shrub of the Labiate species which grows largely in the Canary Islands. It forms a bright yellow oil with an odour recalling that of pennyroyal. Its constants are: specific gravity at 15°, 0·9248; rotatory power, +2° 57′; acid value, 0; saponification value, 11·1; after acetylization, 58·83; coefficient of refraction, 1·48229; solubility, 2·5 volumes of 70 per cent., 0·7 volume of 80 per cent. alcohol. The oil distils mainly between 162° and 234° C. Its chief constituents are pulegone and menthone; it contains but little limonene.

F. H. L.

Detection of Carbazol and Phenanthrene in Anthracene. H. Behrens. (*Rec. trav. chim. des Pays-Bas et de la Belge*, 1902, xxi., 252; through *Chem. Zeit. Rep.*, 1902, 266.)—To detect carbazol, anthracene is extracted with cold acetic ether and the liquid evaporated to dryness. The residue is again taken up in a few drops of the same solvent, and allowed to evaporate on a watch-glass. The carbazol separates out at the edges, the anthracene in the middle of the mass. A little of the former portion is removed with a needle, and treated with a small drop of nitrobenzene in which a few little crystals of phenanthrene-quinone have been suspended. In the presence of only 0·5 per cent. of carbazol copper-coloured crystalline plates are produced, pure anthracene giving no reaction. To detect phenanthrene, the anthracene is extracted with benzene and the solid portion of the extract treated with nitrobenzene containing some *a*-dinitrophenanthrene-quinone. If the original anthracene contained only 1 per cent. of phenanthrene, the existence of the latter will be shown by the formation of the characteristic brown rod-like crystals of the condensation product.

F. H. L.

The Extraction of Alkaloids from Alkaline Solutions. E. Springer. (*Pharm. Zeit.*, 1902, xlvii., 82, 83; through *Zeit. für Untersuch. der Nahr. und Genussmittel*, 1902, v., 935, 936.)—By employing an apparatus in which aqueous

solutions may be extracted continuously with immiscible solvents (see ANALYST, 1892, p. 44), it was found that the whole of the commonly occurring alkaloids can be quantitatively extracted with chloroform in this manner. Morphine, not being readily soluble in pure chloroform, is best extracted with chloroform containing 10 per cent. of alcohol, after first liberating the morphine by ammonia or sodium hydrogen carbonate, as sodium hydroxide is not suitable. In the case of veratrine, codeine or cocaine, an excess of alkali is to be avoided. Sodium hydrogen carbonate should be employed to liberate codeine from its salts, as sodium hydroxide is liable to cause the retention of small traces of the alkaloid in the aqueous solution. Strychnine is rapidly extracted from solutions containing sodium hydroxide, and in traces even from acid solutions. Cocaine is also readily extracted, but it should be noted that it is liable to decomposition at moderately low temperature; chloroform extracts traces of this alkaloid from acid solutions. Traces of atropine and narcotine are also obtained from acid solutions, whilst the extraction is rapid when the solution is alkaline. Coniine and nicotine must be extracted at quite low temperatures. Other solvents, such as isobutyl alcohol, amyl alcohol, benzene, toluene, carbon tetrachloride, and petroleum may, in certain cases, be used instead of chloroform. W. P. S.

The Extraction of Alkaloids from Acid Solutions and Salts of the Alkaloids from Aqueous Solutions. E. Springer. (*Apoth. Zeit.*, 1902, xvii., 225, 226.)—Chloroform was found to extract alkaloids most readily from solutions rendered acid with hydrochloric acid as compared with other acid solutions, and it follows that the hydrochlorides are comparatively more soluble than other salts of the alkaloids. In testing for traces of alkaloids hydrochloric acid solutions should not be shaken out with chloroform, unless these extracts are tested for alkaloids as well as the alkaline extractions. From sulphuric acid and phosphoric acid solutions less alkaloid is removed by chloroform, and in the case of solutions acidified with tartaric acid very little. Only aconitine and narcotine salts are appreciably soluble in chloroform. Codeine, cocaine, quinine, morphine, coniine, and nicotine salts are practically insoluble, and strychnine, atropine, and veratrine only yield a minute trace of soluble salt. Morphine is not dissolved by pure chloroform from acid solutions, but is to some extent soluble in chloroform containing 10 per cent. of alcohol.

Morphine hydrochloride, morphine sulphate, and quinine sulphate are not dissolved by chloroform from neutral solutions; cocaine hydrochloride gives traces of the free base. All the other alkaloids are extracted from neutral solutions, the soluble portion consisting partly of the salt and partly of free base. The more concentrated the solutions are, the more is dissolved by the chloroform. The presence of glycerol, saponin, etc., has no influence on the solubilities. W. P. S.

Detection of Blood Colouring-Matters in Urine. O. Rossel. (*Schweig. Wochschr. Chem. Pharm.*, 1901, xxxix., 557, 558; through *Zeit. für Untersuch. der Nahr. und Genussmittel*, 1902, v., 942.)—The urine is strongly acidified with acetic acid and shaken out with an equal volume of ether. A drop of water is added to the

ethereal extract, and then 15 to 30 drops of old oil of turpentine or 5 to 6 drops of fresh hydrogen peroxide. After shaking, 10 to 20 drops of a freshly-prepared 2 per cent. solution of Barbadoes aloïn in alcohol (70-90 per cent.) are added, and the mixture is again well shaken. In the presence of minute traces of blood—too small to be detected spectroscopically—the aqueous layer becomes distinctly red in colour within three minutes. After ten minutes the colour turns to bright cherry red.

W. P. S.

A New Colorimetric Method of determining Mercury in Urine. Schumacher and W. Jung. (*Zeit. anal. Chem.*, 1902, xli., 461-484.)—As a rule, 500 c.c. of the urine are taken, but if subsequently no coloration is given with hydrogen sulphide, the amount of mercury, if present, cannot exceed 0·6 milligramme per litre; and in such cases a litre or more of the urine should be concentrated on the water-bath, with the addition of a few grammes of sodium chloride to prevent any mercuric chloride volatilizing. The 500 c.c. of urine are heated to the boiling-point with 50 c.c. of concentrated hydrochloric acid and about 5 grammes of potassium chlorate, after which the contents of the flask are cooled to about 80° C., and treated with 12 grammes of pure zinc filings. Eventually an additional 3 grammes of zinc are added, and the flask set aside for two hours. The supernatant liquid is then decanted from the deposit of zinc particles (which will have combined with all the mercury present), and this deposit washed twice with water, then treated for a few minutes with a dilute solution of sodium hydroxide, and again washed twice with water. The residue is now mixed with 50 c.c. of dilute hydrochloric acid and a little potassium chlorate, and heated gently over a small flame until everything has dissolved, more potassium chlorate being added from time to time. The solution is cooled to 70° to 80° C., mixed with about 5 c.c. of alcohol, again boiled, cooled, and transferred to a 100 c.c. flask. The liquid is mixed with a few c.c. of hydrogen sulphide water and made up to the mark, and the colour compared with that given by a freshly-prepared standard solution of mercuric chloride.

The authors state that this is the most rapid and simple method known, and they show by the results of test experiments that it is extremely accurate, whilst being capable of determining 0·1 milligramme of mercury per litre after concentrating the original urine from 3 litres to 1 litre.

C. A. M.

The Examination of Civet. A. Hébert. (*Bull. Soc. Chim.*, 1902, xxvii., 997-1000.)—The samples of guaranteed purity examined by the author melted at 36° to 37° C.

The principal constituent of civet is soluble in ether, benzene, chloroform, and petroleum spirit, but is only soluble with difficulty in alcohol, methyl alcohol, and acetone at the ordinary temperature. The residue left on treating civet with organic solvents consists of accidental impurities, hair, etc., In three samples examined by the author it ranged from 3·6 to 5·3 per cent.

The ash varied from 0·8 to 1·2 per cent., and consisted of sulphates (in large

quantity), carbonates, phosphates, and other salts of iron, aluminium, calcium, magnesium, potassium, and sodium. Chlorides were almost entirely absent. The rotatory power of a filtered solution in a mixture of alcohol and ether was practically nil.

When civet is distilled in a current of steam nearly the whole of the malodorous part (skatole) passes over, whilst the residue has a musk-like odour, and resembles fat in appearance. Civet is not readily saponified, and the boiling with alcoholic alkali must be continued for a very long time. When the saponification product is taken up with warm water an opalescent solution is obtained, and on acidulating this with sulphuric acid a layer of fatty acids rises to the surface. In the three samples examined by the author these melted at 39° C., and amounted to from 51 to 70 per cent. of the original material.

The composition of civet can be considerably influenced by the method of extraction. In some cases the instrument used for the incision is smeared with honey, and the civet then reduces Fehling's solution, and has a rotatory power. Fat is also commonly used to smear the instrument, and in the author's opinion this was the case with the samples examined by him. C. A. M.

INORGANIC ANALYSIS.

Technical Estimation of Silver Bromide in Photographic Dry Plates. V. Bellach. (*Allg. Phot. Zeit.*, 1902, ix., 165; through *Chem. Zeit. Rep.*, 1902, 276.)—The film is brought into a porcelain basin and treated in the cold with a 1 : 20 solution of potassium cyanide for about fifteen minutes. When all the silver has been dissolved the liquid is rinsed into a beaker, and warmed with strong nitric acid to drive off the hydrocyanic acid. The silver bromide is thus reproduced, and coheres into a mass which can easily be washed by decantation. Finally, it is thrown on a tared double paper, washed thoroughly, dried at 110° or 120° C., and weighed. Alternatively, the cyanide solution may be electrolysed with a current of 3·7 to 4·1 volts at a density of 0·2 to 0·5 ampère per 100 square centimetres.

F. H. L.

Strong Sulphuric Acid as a Solvent in the Analysis of Tin Alloys. H. Nissenson and F. Crotogino. (*Chem. Zeit.*, 1902, xxvii., 984.)—This is essentially a continuation of the authors' former paper (Analyst, xxvii., 336). In the absence of lead 0·5 gramme of the sample is heated with 7 c.c. of strong sulphuric acid for a few minutes till solution is complete. The liquid, which contains the tin, is cooled, cautiously diluted with hot water, and the yellow precipitate, comprising all the tin and antimony (the latter in the lower state of oxidation), is allowed to settle in a hot place. As it is, it filters better than the similar precipitate obtained after treatment with nitric acid, but it may be improved in this respect by oxidation, preferably by means of ammonium persulphate. After washing, it is strongly ignited with the filter-paper, and weighed as $SnO_2 + SbO_2$.* The filtrate is raised to the boiling-point and the

* [Ignition of a mixture of tin and antimony oxides with the filter-paper causes loss by volatilization.—B. B.]

copper is thrown down with thiosulphate; in the next filtrate iron, cadmium, etc., are determined. A second sample is dissolved as before, and the solution is diluted with a little water and 15 c.c. of weak hydrochloric acid. The antimony (with any copper) is precipitated with iron wire, dissolved in aqua regia, thrown down with sulphuretted hydrogen, dissolved in sodium sulphide, and the antimony itself is recovered by electrolysis. The insoluble metallic sulphides are taken up in nitric acid, and the liquid is examined for copper, bismuth, etc. If no iron and but little copper are present the antimony can be estimated by treating the original solution with hydrochloric and tartaric acids and titrating with iodine, or Györy's direct process with potassium bromate may be employed. If a volumetric method is decided upon, the sulphuric acid must be cooled immediately the alloy is dissolved, or some of the antimony may be oxidized.

In the presence of lead and tin only, the sulphuric acid solution is diluted with hot water, and a considerable quantity of ammonium oxalate is added. After cooling the lead sulphate is filtered off, and the filtrate is electrolysed with a current of 1 to 1·5 ampères per 100 square centimetres to recover the tin—such being the method for soft solders. If other metals are expected, the first solution is diluted with hot water only, the residue washed with weak sulphuric acid, ignited and weighed as $PbSO_4 + SnO_2 + SbO_2$. In the filtrate, copper, iron, cadmium, zinc, etc., are determined as usual. A second sample is treated identically, but the precipitate of lead, antimony, and tin is dissolved in hot dilute hydrochloric acid, two drops of sulphuric acid are introduced, the whole is cooled, and the lead is filtered off. In the filtrate antimony is thrown down with iron wire as before, and the tin either estimated by difference or by precipitation with sulphuretted hydrogen after removal of the elemental antimony. If the solution containing iron is evaporated to free it from hydrochloric acid till fumes of sulphuric acid appear, the tin will not be thrown down quantitatively by mere dilution with water, and the precipitate which is obtained will be contaminated with much iron.

The method is not suitable for the analysis of alloys (bronze and the like) containing much copper, as the dissolution does not proceed rapidly enough.

F. H. L.

Photometric Determination of Iron. J. I. D. Hinds and Myrtis Louise Cullum. (*Journ. Amer. Chem. Soc.*, xxiv., 848.)—This paper constitutes the first application of the photometric method to the case of coloured precipitates. For the readings a candle and simple photometric cylinder were used (*Journ. Amer. Chem. Soc.*, xviii., 661). The iron was precipitated in a solution containing up to 1 per cent. of nitric acid by means of a 5 per cent. solution of potassium ferrocyanide. Since ferric ferrocyanide is soluble in excess of potassium ferrocyanide, the addition of the latter to the iron solution must be made drop by drop, after a precipitate has once appeared, a reading being taken between each addition of ferrocyanide, and the lowest one being taken, since this represents the maximum opacity.

If y is the percentage of iron in the solution and x the photometric reading, y may be calculated from the empirical formula

$$y = \frac{0{\cdot}03027}{x - 0{\cdot}28}.$$

Using this formula, results were obtained in a series of test analyses correct to 1 part of iron in 1,000,000 of solution, or, with the quantities of iron used, which were generally less than 0·01 gramme, correct to about 1 per cent. on the iron.

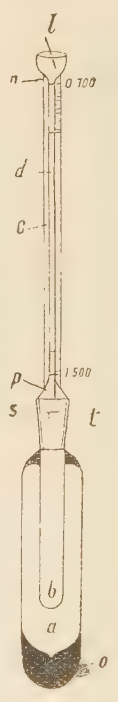
A. G. L.

The Precipitation of Ammonium Vanadate by means of Ammonium Chloride. Arthur Rosenheim. (*Zeits. anorg. Chem.*, xxxii., 181.)—In this reply to Gooch and Gilbert's vindication of Gibbs's method (*supra*), the author states that they appear to have misunderstood his original paper, as he himself obtained sufficiently accurate results when working with pure vanadium solutions, and that his strictures of the method only applied to the case in which tungstic as well as vanadic acid is present, as then not inconsiderable amounts of ammonium tungstate are carried down with the ammonium vanadate. The other investigators mentioned by Gooch and Gilbert also only criticised the method as a means of separating vanadic from molybdic acid.

A. G. L.

APPARATUS.

A New Areo-pyknometer. P. N. Raikow. (*Chem. Zeit.*, 1902, xxvi., 704.)—The apparatus shown in the accompanying diagram possesses several advantages over the areo-pyknometers already devised by Eichhorn and by Vandenvyver, inasmuch as the temperature of the liquid under investigation need not be the same when it is inserted in the bulb as when its density has to be determined. Also it is possible to determine the specific gravity of the liquid on the same portion thereof at different temperatures in succession. The pyknometer is constructed in two parts —a lower, *o t*, and an upper, *p l*. *o* is the usual weight of mercury or shot, *a* is an empty space, and *b* is the liquid reservoir. The upper part consists of a pair of concentric tubes, *d* and *c*, the smaller of which is 1 millimetre in diameter, joined together at their ends, and containing in the annulus the ordinary pyknometer scale. At their base the united tubes become the stopper *p*, through which *d* passes, terminating in a cone to prevent entanglement of air. The stopper is given nearly parallel sides, is about 15 millimetres long, and is very carefully ground in. At the top the inner tube *d* expands into the funnel *l*, which holds about 0·5 c.c. The reservoir *b* is filled with the liquid to the mark *s*, which is in such a position that when the upper part is inserted in the neck the said liquid reaches slightly above the point *n*. The temperature of the whole is then adjusted, and the surplus liquid (that above *n*) is removed with a small pipette or a piece of porous paper. After immersing the apparatus in distilled water and taking the reading, the temperature of the liquid may be raised by placing it in warmer water, removing, as before, the surplus fluid; while if the second reading is to be made at a lower temperature than the first, a drop or two of liquid is added to the funnel to make good the con-



traction. By altering the size of the reservoir, or by omitting it altogether, the apparatus may be made suitable for investigating any desired quantity of liquid.

F. H. L.

The Cause of the Destruction of Platinum Crucibles in Phosphate Analysis. W. C. Heraeus. (*Zeits. f. angew. Chem.*, 1902, xxxvii., 917.)—The destructive agent is phosphorus, which may be liberated under certain circumstances by the reduction of the magnesium phosphate. The author found by experiment that carbon causes the reduction at 950° C., and reducing gases—especially hydrogen—act at a considerably lower temperature. An even more dangerous reducing agent is ammonia. It is very important that this substance be driven off at a moderated temperature before raising the crucible to red heat. Ammonia acts far more energetically on the magnesium phosphate if the latter contain free ammonium phosphate, which is liable to be carried down by the precipitate when ammonia and phosphoric acid are present in excess. This condition of things may often occur at the moment of precipitation, but if the precipitate be allowed to stand some time before filtering, the ammonium phosphate will go into solution again. If the precipitate be dried before being ignited, the drying should be carried out at 100° C., for at 130° or 150° a change takes place whereby phosphoric acid is set free. A. M.

REVIEW.

Cane-Sugar and the Process of its Manufacture in Java. By H. C. Prinsen Geerligs, Director of the West Java Sugar Experiment Station. Second Edition. (Office of *Cane-Sugar*. Altrincham. Price 5s.)

This is a small, well-written book of about 100 pages, by a most competent author, and, although one would expect from its title and size that its contents would be very limited in their scope, it will be found to supply valuable information useful to chemists engaged in cane-sugar factories in localities other than Java. Besides giving a clear and concise account of the mode of manufacture, there is a large amount of information respecting the composition of the sugar-cane and the various products derived therefrom, and in addition other data of practical importance to the factory chemist. The contents of the book are divided into four parts:

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B. E. R. N.

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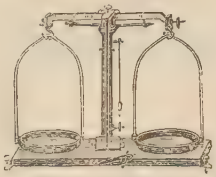

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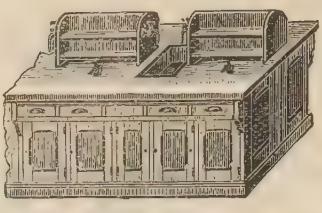

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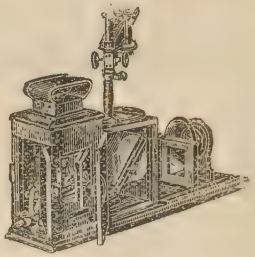

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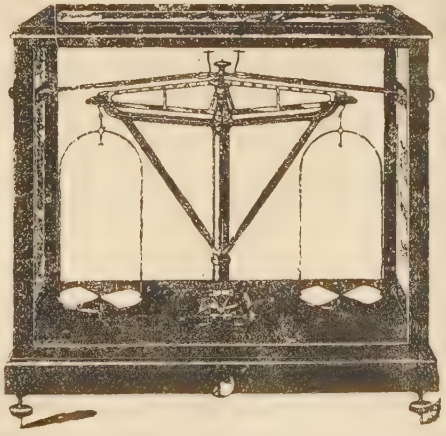

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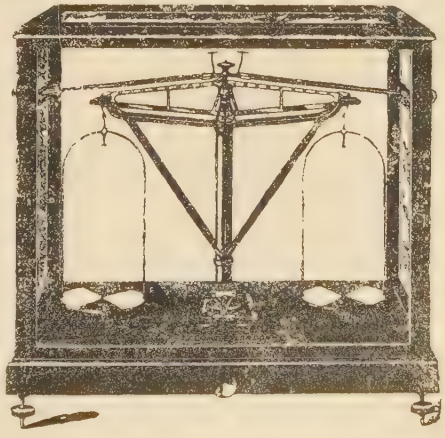

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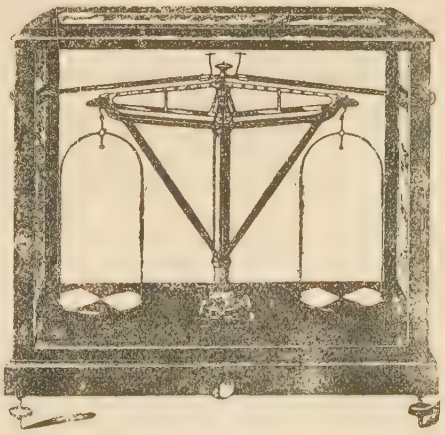

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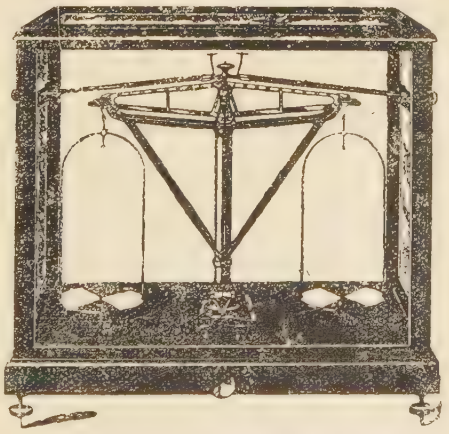

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By order of the Council,

RICHARD B. PILCHER, Registrar and Secretary.

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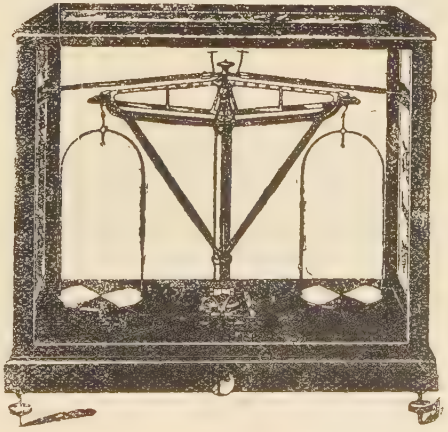

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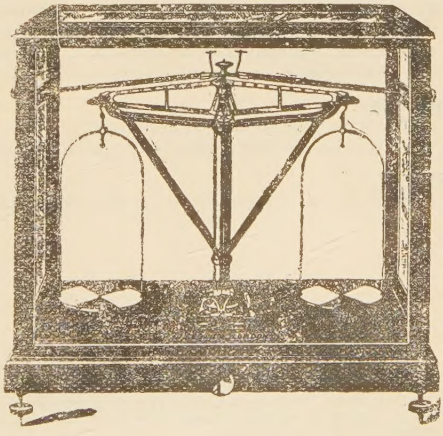

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